भारतीय मानक
Indian Standard

IS 326 (Part 13) : 2020

प्राकृतिक और संश्लेषित सुगंध सामग्री के नमूने लेने और परीक्षण की पद्धतियाँ

भाग 13 1,8-साइनओल अंश ज्ञात करना

(चौथा पुनरीक्षण)

Methods of Sampling and Test for Natural and Synthetic Perfumery Materials

Part 13 Determination of 1,8-Cineole Content

(Fourth Revision)

ICS 71.100.60

© BIS 2020

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

January 2020 **Price Group 2**

Fragrance and Flavour Sectional Committee, PCD 18

FOREWORD

This Indian Standard (Part 13) (Fourth Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Fragrance and Flavour Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1952 and subsequently revised in 1968, 1981 and 2005. Third revision was aligned with ISO 1202 : 1981 'Essential oils — Determination of 1,8-cineole content' under dual numbering system. ISO has withdrawn ISO 1202 : 1981. However, this test method is extensively being used by Indian industry. Therefore, the Committee decided to revise this standard to remove dual numbering with International Standard, ISO 1202 : 1981.

Further, parallel to this method, gas chromatographic method for the determination of 1,8-cineole in essential oils will be the subject of future Indian standards.

In reporting the result of a test or analysis made in according with this standard, if the final value observed or calculated, is to be rounding off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 13 DETERMINATION OF 1,8-CINEOLE CONTENT

(Fourth Revision)

1 SCOPE

This standard (Part 13) specifies a method for the determination of the content of 1,8-cineole in essential oils, the principal constituents of which are 1,8-cineole and terpene hydrocarbons.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
326 (Part 1) : 2018	Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*third revision*)
326 (Part 23) : 2017	Methods of sampling and test for natural and synthetic perfumery materials: Part 23 Preparation of test samples

3 PRINCIPLE

The crystallization temperature of a mixture of essential oil and *o*-cresol is measured which depends on the 1,8-cineole content of the essential oil.

4 REAGENTS

4.1 *o*-Cresol

Purified, anhydrous, melting-point not less than 30.5°C.

4.1.1 As this reagent is hydroscopic, it should be stored in small, well-stoppered bottles, or preferably in sealed flasks. These containers should also be protected from light.

4.1.2 When the *o*-cresol is not in the condition specified above, it is possible to purify it as follows:

Melt a quantity of *o*-cresol (analytical reagent grade), add 5 percent of its mass of distilled water, and allow to crystallize at a temperature of 20 to 25°C. Drain the crystals, and transfer them to a flask fitted with a fractionating column. Distil the first 10 percent (*v/v*) and discard it. Replace the column by a similar one, but dry, and distil 80 percent (*v/v*), the residue in the flask being discarded. Allow the main fraction to crystallize. If its melting point is still below 30.5°C, repeat the distillation as before, as many times as is necessary to obtain a product having a melting point not less than 30.5°C, which is colourless on melting.

4.2 1,8-Cineole, Analytical Reagent Grade

The purity of cineole shall be checked, for example, by measurement of the refractive index at 20°C, which shall be between 1.4550 and 1.4600.

4.3 1,8-Cineole-*o*-cresol Complex

Prepared by mixing equimolecular proportions (in the ratio 154.24/108.13) of the cineole **(4.2)** and the *o*-cresol **(4.1)**, and purified by crystallization from light petroleum (of analytical grade), of distillation range between 40 and 60°C. The crystallization point of the complex shall not be below 55.2°C.

5 APPARATUS

5.1 Calibrated Thermometers

Any standard thermometer of range between 20 and 60°C and accuracy of 0.1°C shall be used.

5.2 Test Tube

About 20 mm diameter and 100 mm long.

5.3 Stout-walled Test Tube

About 30 mm diameter and 125 mm long.

5.4 Apparatus Assembly for Determination of Freezing Point

See Fig. 1, which is given as an example.

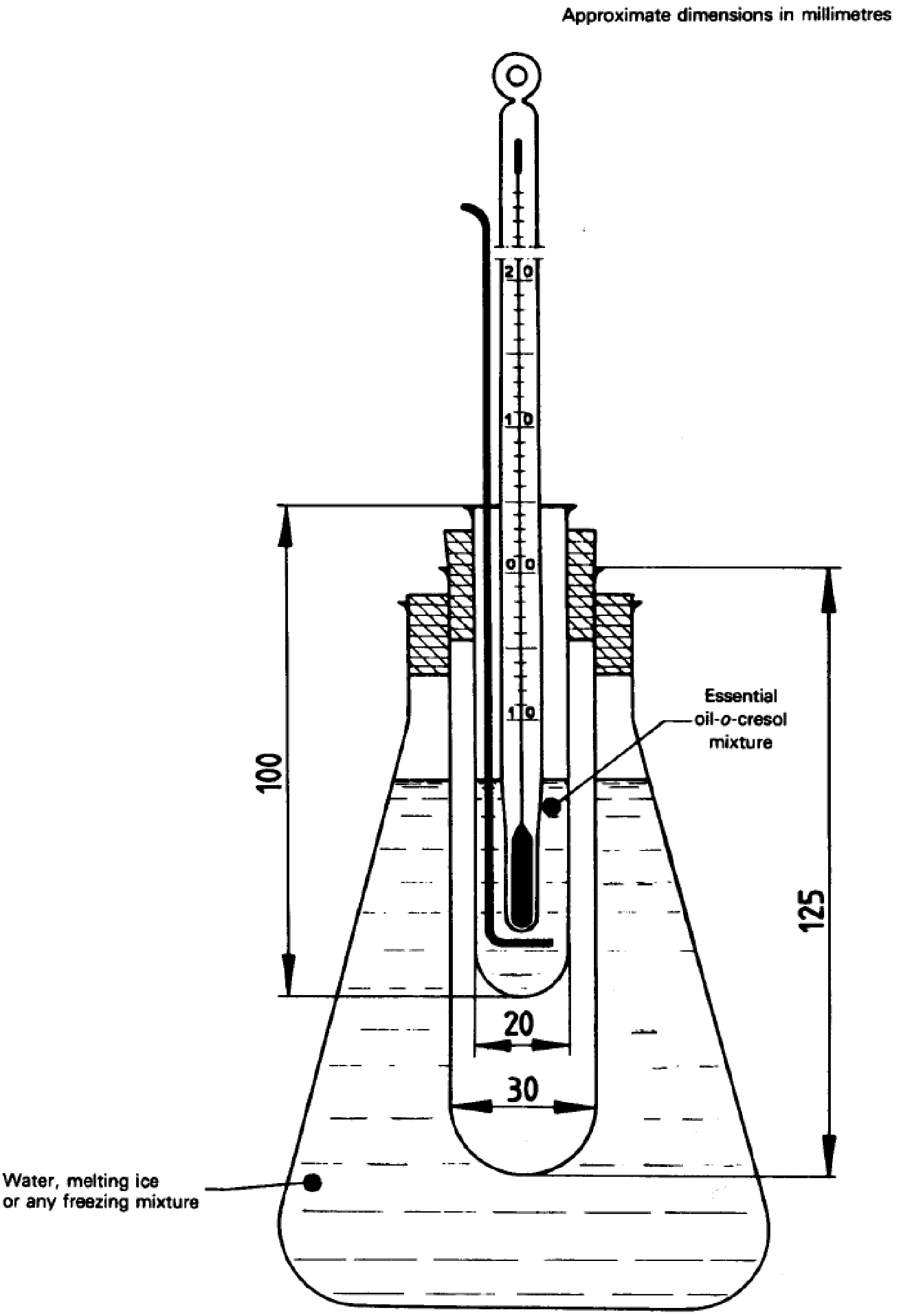
Approximate dimensions in millimetres
2 0
1 0
0 0
1 0
100
125
Essential oil-*o*-cresol mixture
20
30
Water, melting ice or any freezing mixture

FIG. 1 EXAMPLE OF APPARATUS ASSEMBLY FOR DETERMINATION OF FREEZING POINT

5.4.1 It consists of a wide-mouthed jar or bottle of about 500 ml capacity, provided with a bored cork or rubber stopper into which the stout-walled test tube **(5.3)** is inserted. The test tube **(5.2)** is fitted into the stout walled test tube **(5.3)** by means of another bored cork or rubber stopper.

5.4.2 If necessary, the above mentioned vessel may be filled with cold water for cooling prior to the preliminary test (*see* **7.2**) and to the actual determination (*see* **7.3**).

5.4.3 The thermometer **(5.1)** is inserted into the test tube **(5.2)** so that it is located at the centre of the liquid.

5.5 Water Bath

5.6 Agitator

6 SAMPLING

Representative samples of the test material shall be drawn from the lots as prescribed in IS 326 (Part 1).

7 PROCEDURE

7.1 Preparation of Test Sample

Test samples shall be prepared as prescribed in IS 326 (Part 23).

7.2 Preliminary Test

7.2.1 Weigh, to the nearest 0.001 g, 3 g of the freshly prepared test sample (*see* **7.1**) in the test tube **(5.2)** carefully dried, and add 2.10 g of the melted *o*-cresol **(4.1)**.

7.2.2 Place the tube in the apparatus **(5.4)** and allow the mixture to crystallize by cooling, stirring with the agitator **(5.6)**.

7.2.3 When crystallization takes place, there is a small increase in temperature. Note the maximum value obtained, t_1.

7.3 Determination

7.3.1 Re-melt the mixture, at a temperature not exceeding (t_1 by more than 5°C, using the water bath **(5.5)**. Place the test tubes **(5.2)** into the apparatus **(5.4)** maintained at a temperature 5°C below t_1, checking with the thermometer **(5.1)**.

7.3.2 When crystallization begins, or when the temperature of the mixture has fallen to a value 3°C below t_1, stir continuously by means of the agitator **(5.6)**. Take care that the bulb of the thermometer is always completely immersed. Induce the crystallization by rubbing the wall of the test tube with the bulb of the thermometer. Note the maximum temperature at which the mixture crystallizes, t_2.

7.3.3 Repeat the determination until the two highest values obtained for t_2 do not differ by more than 0.2°C.

7.3.4 If super-cooling occurs, induce the crystallization by adding a small crystal of the 1,8-cineole-*o*-cresol complex **(4.3)**.

7.3.5 If t_2 is below 27.4°C, repeat the determination after the addition of 5.10 g of the 1,8-cineole-o-cresol complex **(4.3)**.

8 EXPRESSION OF RESULTS

The content of 1,8-cineole, corresponding to the highest temperature observed (t_2), is given in the table.

If 5.10 g of the 1,8-cineole-*o*-cresol complex **(4.3)** has been added, the 1,8-cineole content of the sample, expressed as a percentage by mass = $2(A - 50)$…….(1)

where ,

A = percentage of 1,8-cineole indicated in the Table 1.

The results shall be expressed to two significant figures. The content of 1,8-cineole, corresponding to the highest temperature observed (t_2), is obtained, where necessary, by interpolation from the data in the Table 1.

Table 1 1,8-Cineole Content as a Function of the Crystallization Temperature of the Essential Oil and *o*-Cresol Mixture

(*Clause* 8)

Sl No. (1)	Crystallization Temperature (°C) (1)	1,8-Cineole Content Percent (*m*/*m*) (2)
1)	24	45.5
2)	25	47
3)	26	48.5
4)	27	49.5
5)	28	50.5
6)	29	52
7)	30	53.5
8)	31	54.5
9)	32	56
10)	33	57
11)	34	58.5
12)	35	60
13)	36	61
14)	37	62.5
15)	38	63.5
16)	39	65
17)	40	67
18)	41	68.5

Table 1 (*Concluded*)

Sl No.	Crystallization Temperature (°C)	1,8-Cineole Content Percent (*m*/*m*)
(1)	(1)	(2)
19)	42	70.5
20)	43	72.5
21)	44	74
22)	45	76
23)	46	78
24)	47	80
25)	48	82
26)	49	84
27)	50	86
28)	51	88.5
29)	52	91
30)	53	93.5
31)	54	96
32)	55	99

9 TEST REPORT

The test report shall state the method used and the result obtained. It shall also mention any operating conditions not specified in this standard, or regarded as optional, as well as any circumstances that might have affected the results.

The test report shall include all details required for the complete identification of the sample.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 18 (14069).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	2323 7617 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019	265 0206 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	2832 9295, 2832 7858 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi